

| | | |
|---|--|--|
| FORM PTO-1390 (REV. 1-98) U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE | | ATTORNEY'S DOCKET NUMBER |
| TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371 | | M8540/271078 |
| INTERNATIONAL APPLICATION NO. PCT/GB00/03275 | | U.S. APPLICATION NO. (If known, see 37 CFR 1.5) unknown 10/070595 |
| TITLE OF INVENTION HIGH TEMPERATURE RESISTANT SALINE SOLUBLE FIBRES | | PRIORITY DATE CLAIMED 10 September 1999 (10.09.99) |
| APPLICANT(S) FOR DO/EO/US Gary Anthony JUBB and Jean-Louis MARTIN | | |
| <p>Applicants herewith submit to the United States Designated/Elected Office (DO/EO/US) the following items and other information:</p> <p>1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.</p> <p>2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.</p> <p>3. <input type="checkbox"/> This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 37 (b) and PCT Articles 22 and 39(1).</p> <p>4. <input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.</p> <p>5. <input checked="" type="checkbox"/> A copy of the International Application as published (35 U.S.C. 371(c)(2))</p> <p>a. <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau).</p> <p>b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau.</p> <p>c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</p> <p>6. <input type="checkbox"/> A translation of the published International Application into English (35 U.S.C. 371(c)(2)).</p> <p>7. <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))</p> <p>a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau).</p> <p>b. <input type="checkbox"/> have been transmitted by the International Bureau.</p> <p>c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</p> <p>d. <input checked="" type="checkbox"/> have not been made and will not be made.</p> <p>8. <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).</p> <p>9. <input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). (unexecuted)</p> <p>10. <input type="checkbox"/> A translation of the annexes of the International Preliminary Examination Report under PCT Article 36</p> <p>11. <input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.197 and 1.98</p> <p>12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</p> <p>13. <input checked="" type="checkbox"/> A FIRST preliminary amendment.</p> <p><input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment.</p> <p>14. <input type="checkbox"/> A substitute specification.</p> <p>15. <input type="checkbox"/> A change of power of attorney and/or address letter.</p> <p>16. <input checked="" type="checkbox"/> Other items or information:</p> <p>a. Certification Under 37 CFR 1.10</p> <p>I hereby certify that this document is being mailed to Box PCT, Commissioner for Patents, Washington, D.C. 20231, via "Express Mail Post Office to Addressee" on this 8th day of March, 2002, Express Mail Label No. EL209599794US</p> <p><i>Angela M. Rossi</i> Angela M. Rossi</p> <p>b. Form PCT/IB/308</p> <p>c. International Preliminary Examination Report</p> | | |

JC13 Rec'd PCT/PTO 08 MAR 2002

| | | | | |
|--|---|--|------------|--------|
| U.S. APPLICATION NO. (known) (See 37 CFR 1.5) Unknown | INTERNATIONAL APPLICATION NO. PCT/GB00/03275 | ATTORNEY'S DOCKET NUMBER M8540/271078 | | |
| 17. <input checked="" type="checkbox"/> The following fees are submitted BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)): | | CALCULATIONS PTO USE ONLY | | |
| Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2) paid to USPTO and International Search Report not prepared by the EPO or JPO | | \$1,040.00 | | |
| International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO | | \$890.00 | | |
| International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2) paid to USPTO | | \$740.00 | | |
| International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) | | \$710.00 | | |
| International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) | | \$100.00 | | |
| ENTER APPROPRIATE BASIC FEE AMOUNT | | = \$890.00 | | |
| Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)). | | \$130.00 | | |
| CLAIMS | NUMBER FILED | NUMBER EXTRA | RATE | \$ |
| Total claims | 13 | 00 | X \$18.00 | \$.00 |
| Independent claims | 01 | 00 | X \$84.00 | \$.00 |
| MULTIPLE DEPENDENT CLAIM(S) (if applicable) | | | + \$280.00 | \$.00 |
| TOTAL OF ABOVE CALCULATIONS | | = | \$1,020.00 | |
| Reduction of $\frac{1}{2}$ for filing by small entity, if applicable. | | -- | \$.00 | |
| SUBTOTAL | | = | \$1,020.00 | |
| Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)). | | \$.00 | | |
| TOTAL NATIONAL FEE | | = | \$1,020.00 | |
| Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40 per property | | \$.00 | | |
| TOTAL FEES ENCLOSED | | = | \$1,020.00 | |
| | | Amount to be refunded: | \$ | |
| | | charged: | \$ | |
| <p>a. <input checked="" type="checkbox"/> Check in the amount of \$1,020.00 is enclosed.</p> <p>b. <input type="checkbox"/> Please charge my Deposit Account No. 11-0855 in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.</p> <p>c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 11-0855.</p> | | | | |
| NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b) must be filed and granted to restore the application to pending status. | | | | |
| Customer No. 23370 | | | | |
| SEND ALL CORRESPONDENCE TO: | | | | |
| John S. Pratt, Esq. | | SIGNATURE | | |
| KILPATRICK STOCKTON LLP | | Name: Dean W. Russell | | |
| 1100 Peachtree Street, Suite 2800 | | Registration No. 33,452 | | |
| Atlanta, Georgia 30309-4530 | | | | |

IN THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US)

Applicants: Gary Anthony JUBB and Jean-Louis MARTIN

International
Application No.: PCT/GB00/03275

U.S. Serial No.:

International
Filing Date: 24 August 2000 (24.08.00)

U.S. Filing Date: 08 March 2002 (08.03.02)

For: HIGH TEMPERATURE RESISTANT
SALINE SOLUBLE FIBRES

Box PCT
Commissioner for Patents
Washington, D.C. 20231

Attorney Docket No. M8540/2710780
Date: 08 March 2002

PRELIMINARY AMENDMENT

Sir:

Kindly amend the above-identified patent application prior to examination:

In the Specification

On page 1, immediately following the title "High Temperature Resistant Saline Soluble Fibres" kindly insert the following paragraph:

--This application claims priority to Great Britain Application No. 9921504.8 filed on September 10, 1999 and Great Britain Application No. 9924867.6 filed on October 20, 1999 and International Application No. PCT/GB00/03275 filed on August 24, 2000 and published in English as International Publication Number WO 01/19744 A1 on March 23, 2001, the entire contents of which are hereby incorporated by reference.--

Express Mail Label No. EL209599794US
U.S. National Phase Entry of PCT/GB00/03275
Filed: 08 March 2002
PRELIMINARY AMENDMENT

In the claims

4. (Amended) A fibre as claimed in claim 1 and comprising:-

CaO > 19 wt%.

6. (Amended) A fibre as claimed in claim 1 and comprising:-

MgO > 14.25 wt%.

9. (Amended) A fibre as claimed in claim 1 and comprising:-

MgO < 16 wt%.

10. (Amended) A fibre as claimed in claim 1 and comprising:-

SiO₂ 65 +/- 0.5 wt%
CaO 20 +/- 0.5 wt%
MgO 15 +/- 0.5 wt%.

11. (Amended) A fibre as claimed in claim 1 and comprising:-

SiO₂ 64.5 – 64.7 wt%
CaO 19.5 – 20.2 wt%
MgO 15.5 – 15.6 wt%.

12. (Amended) A fibre as claimed in claim 1 and comprising:-

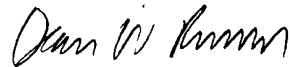
SiO₂ about 65 wt%
CaO about 19.5 wt%
MgO about 15.5 wt%.

Express Mail Label No. EL209599794US
U.S. National Phase Entry of PCT/GB00/03275
Filed: 08 March 2002
PRELIMINARY AMENDMENT

13. (Amended) A fibre as claimed in claim 1 and consisting essentially of:-

CaO 18.7 to 20.2 wt%
MgO 14.47 to 15.9 wt%
SiO₂ 64.5 to 65.1 wt%
Al₂O₃ 0 to 0.56 wt%.

Respectfully submitted,



Dean W. Russell
Reg. No. 33,452

KILPATRICK STOCKTON LLP
1100 Peachtree Street, Suite 2800
Atlanta, Georgia 30309
(404) 815-6528

Express Mail Label No. EL209599794US
U.S. National Phase Entry of PCT/GB00/03275
Filed: 08 March 2002
PRELIMINARY AMENDMENT

Version with markings to show changes made

4. (Amended) A fibre as claimed in [any of] claim[s] 1 [to 3] and comprising:-

CaO > 19 wt%.

6. (Amended) A fibre as claimed in [any of] claim[s] 1 [to 4] and comprising:-

MgO > 14.25 wt%.

9. (Amended) A fibre as claimed in [any preceding] claim 1 and comprising:-

MgO < 16 wt%.

10. (Amended) A fibre as claimed in [any preceding] claim 1 and comprising:-

SiO₂ 65 +/- 0.5 wt%

CaO 20 +/- 0.5 wt%

MgO 15 +/- 0.5 wt%.

11. (Amended) A fibre as claimed in [any of] claim[s] 1 [to 9] and comprising:-

SiO₂ 64.5 – 64.7 wt%

CaO 19.5 – 20.2 wt%

MgO 15.5 – 15.6 wt%.

12. (Amended) A fibre as claimed in [any preceding] claim 1 and comprising:-

SiO₂ about 65 wt%

CaO about 19.5 wt%

MgO about 15.5 wt%.

13. (Amended) A fibre as claimed in [any preceding] claim 1 and consisting essentially of:-

CaO 18.7 to 20.2 wt%

MgO 14.47 to 15.9 wt%

SiO₂ 64.5 to 65.1 wt%

Al₂O₃ 0 to 0.56 wt%.

10/070595

WO 01/19744

PCT/GB00/03275
Rec'd PCT/PTO 08 MAR 2002

HIGH TEMPERATURE RESISTANT SALINE SOLUBLE FIBRES

This invention relates to high temperature resistant saline soluble fibres and particularly relates to calcium-magnesium-silicate fibres.

Calcium-magnesium-silicate fibres are known for example from WO89/12032, WO93/15028 and WO94/15883.

WO89/12032 first disclosed a broad class of fire-resistant saline soluble fibres.

WO93/15028 showed that a class of the fibres of WO89/12032 were usable at temperatures up to 1000°C or more.

WO94/15883 showed that some of the fibres of WO93/15028 had higher use temperatures still, of up to 1260°C or more, and indicated that such fibres needed a SiO₂ excess (defined as the amount of SiO₂ remaining after crystallisation of CaO, MgO and any ZrO₂ as silicates) of greater than 21.8mol%.

WO97/16386 while falling in the general class of calcium-magnesium-silicate fibres looked to low calcium fibres to show use temperatures of 1260°C or more.

It is apparent that there are regions of the CaO-MgO-SiO₂ and CaO-MgO-SiO₂-ZrO₂ composition fields within which high temperature performance fibres can be made and other regions where they cannot.

The applicants have now found a new and narrow range of compositions that are usable at temperatures of 1200°C or more and even 1250°C or 1260°C or more, and yet fall outside the scope of WO94/15883 and WO97/16386. These compositions preferably have little or no zirconia.

Accordingly the present invention provides a fibre having a maximum use temperature of 1200°C or more in which the amount of MgO in mol% is greater than the amount of CaO in mol % and which comprises:-

$\text{SiO}_2 > 64.25 \text{ wt\%}$

$\text{CaO} > 18 \text{ wt\%}$

$\text{MgO} < 17 \text{ wt\%}$.

However, no claim is made to fibres having a SiO_2 excess as specified of greater than 21.8mol%.

Further features of the invention are apparent from the appended claims.

The excess SiO_2 figure is calculated by treating all of the CaO as being bound as $\text{CaO} \cdot \text{MgO} \cdot 2\text{SiO}_2$; all of the ZrO_2 as being bound as $\text{ZrO}_2 \cdot \text{SiO}_2$; and the remaining MgO as being bound as $\text{MgO} \cdot \text{SiO}_2$. The applicants also assume that any Al_2O_3 crystallises as $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$. Any remaining SiO_2 is called the excess SiO_2 .

The invention is illustrated by way of example in the following description with reference to the drawings in which

Fig 1 is a graph showing linear shrinkage with temperature for blankets comprising the fibres A4-2 and A4-3 of Table 1 below.

Fig. 2 is a graph showing shrinkage through the height of blankets comprising the fibres A4-2 and A4-3 of Table 1 below.

Fig. 3 is a graph showing shrinkage of preforms produced from fibres A4-1, A4-2 and A4-3 of Table 1 below.

Table 1 shows compositions extracted from WO89/12032, WO93/15028, WO94/15883, and WO97/16386 together with A4, a target composition fibre having the composition:-

SiO₂ 65 wt%

CaO 19.5 wt%

MgO 15.5 wt%

and A4-1, A4-2, and A4-3, which are analysed fibre samples.

The fibres extracted from the data of WO89/12032 (referred to as Manville fibres), WO93/15028, WO94/15883, and WO97/16386 (referred to as Unifrax fibres) are those for which the SiO₂ excess as specified is less than than 21.8mol% and for which the amount of MgO in mol% is greater than the amount of CaO in mol%.

A4-1 was produced as bulk fibre; A4-2 was produced as needled blanket having a density of approximately 96 kg.m⁻³; and A4-3 was produced as needled blanket having a density of approximately 128 kg.m⁻³.

In Table 1 shrinkages are indicated from the documents concerned or, for A4-1, A4-2, and A4-3, from measuring the shrinkage of vacuum formed preforms of the fibres concerned.

Table 1

| Fibre | Shrinkage at 1260°C | Composition w-% | | | | | | | Composition mol% | | | SiO ₂ Excess | |
|-------------|---------------------|-----------------|-------|------------------|--------------------------------|------------------|------------------|--------|------------------|-------|------------------|--------------------------------|-------|
| | | CaO | MgO | ZrO ₂ | Al ₂ O ₃ | TiO ₂ | SiO ₂ | Others | CaO | MgO | ZrO ₂ | Al ₂ O ₃ | |
| 924 | | 19.78 | 14.54 | 0.66 | 2.57 | | 61.32 | | 19.99 | 20.44 | 0.30 | 1.43 | 57.84 |
| | | | | | | | | | | | | | 15.68 |
| SW-A1 | | 20.50 | 15.20 | | 1.10 | | 63.70 | | 20.16 | 20.79 | 0.00 | 0.59 | 58.46 |
| | | | | | | | | | | | | | 16.92 |
| B5 | 6.00 | 19.90 | 15.10 | 0.10 | 0.20 | | 64.20 | 0.40 | 19.71 | 20.80 | 0.05 | 0.11 | 59.34 |
| | | | | | | | | | | | | | 18.68 |
| 757 | | 20.92 | 15.22 | 0.00 | 0.20 | | 62.60 | | 20.79 | 21.04 | 0.00 | 0.11 | 58.06 |
| | | | | | | | | | | | | | 16.13 |
| A4-1 | 1.86 | 20.20 | 15.50 | | 0.26 | | 64.60 | 0.07 | 19.76 | 21.10 | 0.00 | 0.14 | 59.00 |
| | | | | | | | | | | | | | 18.00 |
| A4-2 | 3.25 | 20.20 | 15.50 | | 0.30 | | 64.50 | 0.06 | 19.78 | 21.11 | 0.00 | 0.16 | 58.95 |
| | | | | | | | | | | | | | 17.89 |
| A4-3 | 2.76 | 20.20 | 15.60 | | 0.28 | | 64.70 | 0.07 | 19.72 | 21.18 | 0.00 | 0.15 | 58.95 |
| | | | | | | | | | | | | | 17.9 |
| A4 target | | 19.50 | 15.50 | | | | 65.00 | | 19.17 | 21.20 | 0.00 | 0.00 | 59.64 |
| | | | | | | | | | | | | | 19.27 |
| SW-A2 | | 21.40 | 15.40 | | 0.80 | | 60.80 | | 21.40 | 21.42 | 0.00 | 0.44 | 56.74 |
| | | | | | | | | | | | | | 13.49 |
| SW-A | | 20.50 | 15.50 | | 3.30 | | 59.30 | | 20.66 | 21.73 | 0.00 | 1.83 | 55.78 |
| | | | | | | | | | | | | | 11.56 |
| 932 | | 21.60 | 15.65 | 0.11 | 1.50 | | 59.85 | | 21.58 | 21.75 | 0.05 | 0.82 | 55.80 |
| | | | | | | | | | | | | | 11.60 |
| Manville104 | | 17.70 | 16.30 | | 1.83 | | 64.10 | | 17.49 | 22.40 | 0.00 | 0.99 | 0.00 |
| | | | | | | | | | | | | | 59.11 |
| B9 | 5.00 | 18.10 | 17.10 | 0.10 | 0.20 | | 64.40 | 0.20 | 17.72 | 23.29 | 0.04 | 0.11 | 58.84 |
| | | | | | | | | | | | | | 17.69 |
| 971 | | 23.92 | 17.36 | 0.05 | 0.74 | | 56.82 | | 23.56 | 23.79 | 0.02 | 0.40 | 52.23 |
| | | | | | | | | | | | | | 4.47 |
| B8 | 6.90 | 18.80 | 17.90 | 0.30 | 0.20 | | 63.00 | 0.20 | 18.30 | 24.24 | 0.13 | 0.11 | 57.23 |
| | | | | | | | | | | | | | 14.46 |
| B16 | 4.30 | 15.10 | 18.10 | 0.30 | 0.10 | 0.10 | 66.00 | 0.20 | 14.78 | 24.65 | 0.13 | 0.05 | 0.07 |
| | | | | | | | | | | | | | 60.31 |
| A2-12 | | 16.55 | 18.00 | 0.05 | 0.33 | | 63.56 | | 16.37 | 24.76 | 0.02 | 0.18 | 58.67 |
| | | | | | | | | | | | | | 17.34 |
| A2-30 | | 16.06 | 18.21 | 0.00 | 0.40 | | 63.68 | | 15.89 | 25.07 | 0.00 | 0.22 | 58.82 |
| | | | | | | | | | | | | | 17.64 |

Table 1

| Fibre | Shrinkage at 1260°C | Composition w% | | | | | | Composition mol% | | | | SiO ₂ Excess | |
|--------------|---------------------|----------------|-------|------------------|--------------------------------|------------------|------------------|------------------|-------|-------|------------------|--------------------------------|-------|
| | | CaO | MgO | ZrO ₂ | Al ₂ O ₃ | TiO ₂ | SiO ₂ | Others | CaO | MgO | ZrO ₂ | Al ₂ O ₃ | |
| A2-23 | | 18.59 | 18.78 | 0.05 | 0.48 | | 60.20 | | 18.37 | 25.82 | 0.02 | 0.26 | 55.53 |
| 71 | | 24.04 | 19.66 | 0.00 | 0.20 | | 54.68 | | 23.44 | 26.67 | 0.00 | 0.11 | 49.77 |
| A2-19 | | 18.48 | 19.74 | 0.00 | 0.54 | | 58.71 | | 18.29 | 27.18 | 0.00 | 0.29 | 54.24 |
| A2-21 | | 13.74 | 19.98 | 0.13 | 0.34 | | 64.16 | | 13.51 | 27.34 | 0.06 | 0.18 | 58.90 |
| Unifrax comp | 13.10 | 0.77 | 16.90 | 34.10 | 0.97 | | 46.40 | | 0.92 | 28.11 | 18.55 | 0.64 | 51.78 |
| 13 | | 13.62 | 22.74 | 0.08 | 0.31 | | 61.38 | | 13.25 | 30.79 | 0.04 | 0.17 | 55.76 |
| A2-24 | | 9.74 | 23.10 | | 2.15 | | 65.10 | | 9.38 | 30.95 | 0.00 | 1.14 | 58.53 |
| Manville105 | | 8.67 | 24.00 | | 0.02 | | 67.20 | | 8.27 | 31.86 | 0.00 | 0.01 | 59.86 |
| Manville79 | | 10.99 | 24.18 | 0.07 | 0.33 | | 62.36 | | 10.66 | 32.64 | 0.03 | 0.18 | 56.48 |
| A2-25 | | 23.40 | 0.89 | 21.70 | 24.10 | 0.90 | 51.90 | | 0.98 | 33.18 | 12.05 | 0.54 | 53.24 |
| Unifrax comp | 14 | | | | | | | | | | | | 6.49 |
| A2-35 | | 8.88 | 24.88 | 0.47 | 0.29 | | 64.12 | | 8.56 | 33.37 | 0.21 | 0.15 | 57.71 |
| Manville78 | | 6.43 | 26.50 | | | | 67.10 | | 6.07 | 34.80 | 0.00 | 0.00 | 59.13 |
| A2-34 | | 6.63 | 26.20 | 0.80 | 0.23 | | 64.85 | | 6.37 | 35.01 | 0.35 | 0.12 | 58.15 |
| Unifrax comp | 15 | | | | | | 55.70 | | 0.80 | 36.82 | 7.98 | 0.43 | 53.96 |
| Manville77 | | 4.02 | 28.70 | | 0.59 | | 66.10 | | 3.79 | 37.68 | 0.00 | 0.31 | 58.22 |
| Unifrax 42 | 2.00 | 0.30 | 29.75 | 0.58 | 0.62 | | 68.63 | | 0.28 | 38.92 | 0.25 | 0.32 | 60.23 |
| | | | | | | | | | | | | | 20.47 |

Table 1

| Fibre | Shrinkage at 1260°C | Composition wt% | | | | | | Composition mol% | | | SiO ₂ Excess | | | | |
|-------------|---------------------|-----------------|-------|------------------|--------------------------------|------------------|------------------|------------------|------|-------|-------------------------|--------------------------------|------------------|------------------|-------|
| | | CaO | MgO | ZrO ₂ | Al ₂ O ₃ | TiO ₂ | SiO ₂ | Others | CaO | MgO | ZrO ₂ | Al ₂ O ₃ | TiO ₂ | SiO ₂ | |
| Manville106 | | 2.70 | 29.70 | | 1.56 | | 65.60 | | 2.54 | 38.94 | 0.00 | 0.81 | 0.00 | 57.71 | 15.41 |
| Manville80 | | 1.60 | 30.10 | | | | 68.40 | | 1.49 | 39.02 | 0.00 | 0.00 | 0.00 | 59.49 | 18.98 |
| Manville71 | | 3.12 | 30.10 | | 1.15 | | 65.40 | | 2.92 | 39.26 | 0.00 | 0.59 | 0.00 | 57.23 | 14.45 |
| Manville76 | | 3.12 | 30.10 | | 1.15 | | 65.40 | | 2.92 | 39.26 | 0.00 | 0.59 | 0.00 | 57.23 | 14.45 |
| Unifrax 47 | 7.70 | 0.25 | 30.26 | 0.01 | 1.86 | | 67.53 | | 0.23 | 39.56 | 0.00 | 0.96 | | 59.24 | 18.48 |
| Unifrax 40 | 9.20 | 0.27 | 30.57 | 0.58 | 0.92 | | 67.52 | | 0.25 | 39.90 | 0.25 | 0.47 | | 59.13 | 18.25 |
| 765 | | 3.90 | 35.07 | 0.00 | 2.12 | | 57.78 | | 3.62 | 45.26 | 0.00 | 1.08 | | 50.04 | 0.07 |

It can be seen that the fibres according to the present invention show lower shrinkage at 1260°C than do the extracted fibres other than fibre Unifrax 42 which has a radically different composition.

In Figs. 1-3, graphs show the shrinkage characteristics of the fibres A4-1, A4-2, and A4-3 after 24 hours exposure to the indicated temperatures. it can be seen that the fibres are readily usable at temperatures of 1200°C or more.

Table 2 below shows the results of solubility tests on the fibres in physiological saline solution indicating that the fibres are soluble in body fluids. (See WO94/15883 for a discussion of methods of measuring solubility). Pairs of results are indicated for separate tests on each sample as is a mean total solubility.

| Fibre type | Solubility (ppm) | | | | |
|------------|------------------|-----|------------------|-------|------------|
| | CaO | MgO | SiO ₂ | Total | Mean Total |
| A4-1 | 102 | 115 | 171 | 388 | 383 |
| | 105 | 110 | 162 | 377 | |
| A4-2 | 105 | 116 | 172 | 393 | 395 |
| | 114 | 117 | 166 | 397 | |
| A4-3 | 114 | 123 | 166 | 403 | 411 |
| | 114 | 128 | 177 | 419 | |

A typical range of compositions for fibres of the present invention would be

SiO₂ 65 ± 0.5 wt%

CaO 20 ± 0.5 wt%

MgO 15 ± 0.5 wt%.

Further tests were made on fibres having the inventive composition of SiO₂ 65%, CaO 19.5%, MgO 15.5% in comparison with Superwool 607TM, a fibre having the nominal composition (by weight) of SiO₂ 65%, CaO 29.5%, MgO 5.5%, and Al₂O₃ <1% ; Superwool 612TM, a fibre having the nominal composition (by weight) of SiO₂ 64.5%, CaO 17%, MgO 13.5%, ZrO₂ 5%; and refractory ceramic fibre having the nominal composition SiO₂ 56%, Al₂O₃ 44%.

The first test was aimed at indicating the amount of dust that might be released on handling. The test comprised the determination of the amount of dust present in a sample of blanket made from the respective fibres. The samples of blanket were vibrated on a Fritsch Analysette type 3010 vibratory sieve shaker, which was set for a frequency of 3000Hz and vertical amplitude of 0.5mm. The apparatus was equipped with a 1.6mm sieve and a pan. In the test method a sample of blanket 135mm x 135mm was placed on the sieve and vibrated for 10 minutes. The material collected in the pan was weighed and expressed as a percentage of the original weight of the sample. The results were as indicated below:-

| <u>Fibre Material</u> | <u>Percent dust released</u> |
|-----------------------------|------------------------------|
| Superwool 607 TM | 0.16% |
| Inventive material | 0.18% |
| Refractory ceramic fibre | 0.25% |
| Superwool 612 TM | 0.36% |

From this it can be seen that the inventive fibre is of comparable low dustiness to Superwool 607TM.

The second test made was to look to the shrinkage behaviour of blanket formed from the inventive fibre and the two SuperwoolTM fibres at high temperatures. Samples of blanket were exposed to specified temperatures for 24 hour periods and their linear shrinkage measured. The results are indicated in Table 3 below:-

Table 3

| Sample | Temperature | | | | | |
|-----------------|-------------|--------|--------|--------|--------|--------|
| | 1050°C | 1110°C | 1150°C | 1200°C | 1250°C | 1300°C |
| Inventive fibre | 0.8 | 0.6 | 1.0 | 1.0 | 1.5 | 4.2 |
| Superwool 612™ | 0.7 | 1.0 | 1.1 | 1.7 | 1.8 | 12.1 |
| Superwool 607™ | 0.4 | 0.4 | 0.5 | 0.6 | 4.8 | 7.8 |

This shows that the inventive fibre is comparable in performance with both Superwool™ fibres up to 1200°C. At 1250°C the Superwool 607™ fibre shows a shrinkage of 4.8% (which would be considered as too high a shrinkage for most applications). At 1300°C the inventive fibre while still showing a high shrinkage of 4.2% is the best of the three fibres tested.

A further series of tests were made to produce fibres on a production scale and the compositions of fibres obtained were as set out in Table 4 below. All showed low shrinkage at 1250°C (shrinkages were measured by different methods to Table 1 and are not directly comparable). In combination with the results of Table 1, this shows samples of usable characteristics having compositions consisting essentially of (in wt%):-

CaO 18.7 to 20.2

MgO 14.47 to 15.9

SiO₂ 64.5 to 65.1

Al₂O₃ 0 to 0.56

The fibres of the present invention therefore have a high solubility (desired to permit fibres to be cleared from the body quickly); a low dustiness (desired to reduce the amount of fibre that can be inhaled); and good high temperature characteristics.

Table 4

| Fibre | Linear shrinkage (%) at 1250°C | Composition wt% | | | | | | Composition mol% | | | | SiO ₂ Excess | | |
|-------|-----------------------------------|-----------------|-------|------------------|--------------------------------|------------------|------------------|------------------|-------|------|------------------|--------------------------------|-------|-------|
| | | CaO | MgO | ZrO ₂ | Al ₂ O ₃ | TiO ₂ | SiO ₂ | Others | CaO | MgO | ZrO ₂ | Al ₂ O ₃ | | |
| SM | 0.80 | 20.10 | 15.00 | 0.06 | 65.00 | | 19.77 | 20.52 | 0.00 | 0.03 | | 59.67 | 19.35 | |
| TCI | 0.78 | 18.90 | 15.50 | 0.38 | 65.10 | | 18.63 | 21.26 | 0.00 | 0.21 | | 59.90 | 19.81 | |
| TCUK | no preform made | 18.70 | 15.90 | 0.44 | 64.80 | | 18.41 | 21.78 | 0.00 | 0.24 | | 59.56 | 19.13 | |
| TCUK2 | 2.6 (measured at 1300°C) | 19.30 | 14.47 | 0.56 | 0.03 | 64.54 | 0.40 | 19.30 | 20.13 | 0.00 | 0.31 | | 60.24 | 20.50 |

CLAIMS

1. A fibre having a maximum use temperature of 1200°C or more which comprises:-

SiO₂ >64.25 wt%

CaO >18 wt%

MgO < 17 wt%

and in which the amount of MgO in mol% is greater than the amount of CaO in mol % and in which the SiO₂ excess as specified is no greater than 21.8mol%..

2. A fibre as claimed in claim 1 and comprising:-

CaO <21 wt%.

3. A fibre as claimed in claim 2 and comprising:-

CaO <20.5 wt%.

4. A fibre as claimed in any of claims 1 to 3 and comprising:-

CaO >19 wt%.

5. A fibre as claimed in claim 4 and comprising:-

CaO > 19.5 wt%

6. A fibre as claimed in any of claims 1 to 4 and comprising:-

MgO > 14.25 wt %.

7. A fibre as claimed in claim 6 and comprising:-

MgO > 14.75 wt %.

8. A fibre as claimed in claim 7 and comprising:-

MgO > 15.25 wt %.

9. A fibre as claimed in any preceding claim and comprising:-

MgO < 16 wt %.

10. A fibre as claimed in any preceding claim and comprising:-

SiO₂ 65 ± 0.5 wt%

CaO 20 ± 0.5 wt%

MgO 15 ± 0.5 wt%.

11. A fibre as claimed in any of claims 1 to 9 and comprising:-

SiO₂ 64.5 - 64.7 wt%

CaO 19.5 - 20.2 wt%

MgO 15.5 - 15.6 wt%.

12. A fibre as claimed in any preceding claim and comprising:-

SiO₂ about 65 wt%

CaO about 19.5 wt%

MgO about 15.5 wt%.

13. A fibre as claimed in any preceding claim and consisting essentially of:-

CaO 18.7 to 20.2wt%

MgO 14.47 to 15.9wt%

SiO₂ 64.5 to 65.1wt%

Al₂O₃ 0 to 0.56wt%

WO 01/19744 A1

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
22 March 2001 (22.03.2001)

PCT

(10) International Publication Number
WO 01/19744 A1

(51) International Patent Classification⁷: **C03C 13/00**

(21) International Application Number: **PCT/GB00/03275**

(22) International Filing Date: 24 August 2000 (24.08.2000)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
9921504.8 10 September 1999 (10.09.1999) GB
9924867.6 20 October 1999 (20.10.1999) GB

(71) Applicant (for all designated States except US): **THE MORGAN CRUCIBLE COMPANY PLC [GB/GB];**
Morgan House, Madeira Walk, Windsor, Berkshire SL4 1EP (GB).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **JUBB, Gary, Anthony [GB/GB];** Morgan Materials Technology Limited, Bewdley Road, Stourport-on-Severn, Worcestershire

(74) Agent: **PHILLIPS & LEIGH**; 5 Pemberton Row, London EC4A 3BA (GB).

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

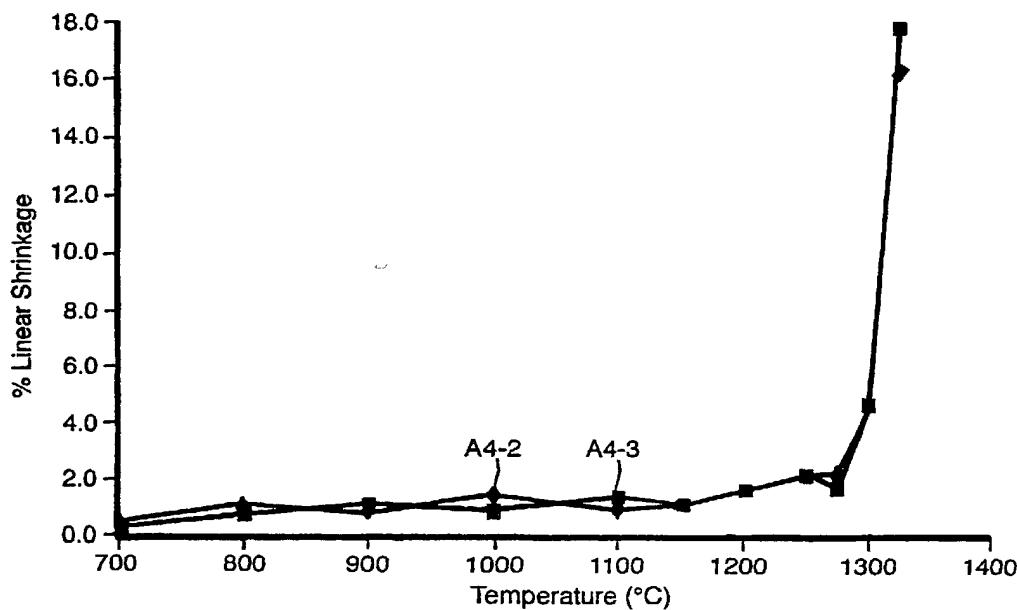
(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

— *With international search report.*

[Continued on next page]

(54) Title: HIGH TEMPERATURE RESISTANT SALINE SOLUBLE FIBRES



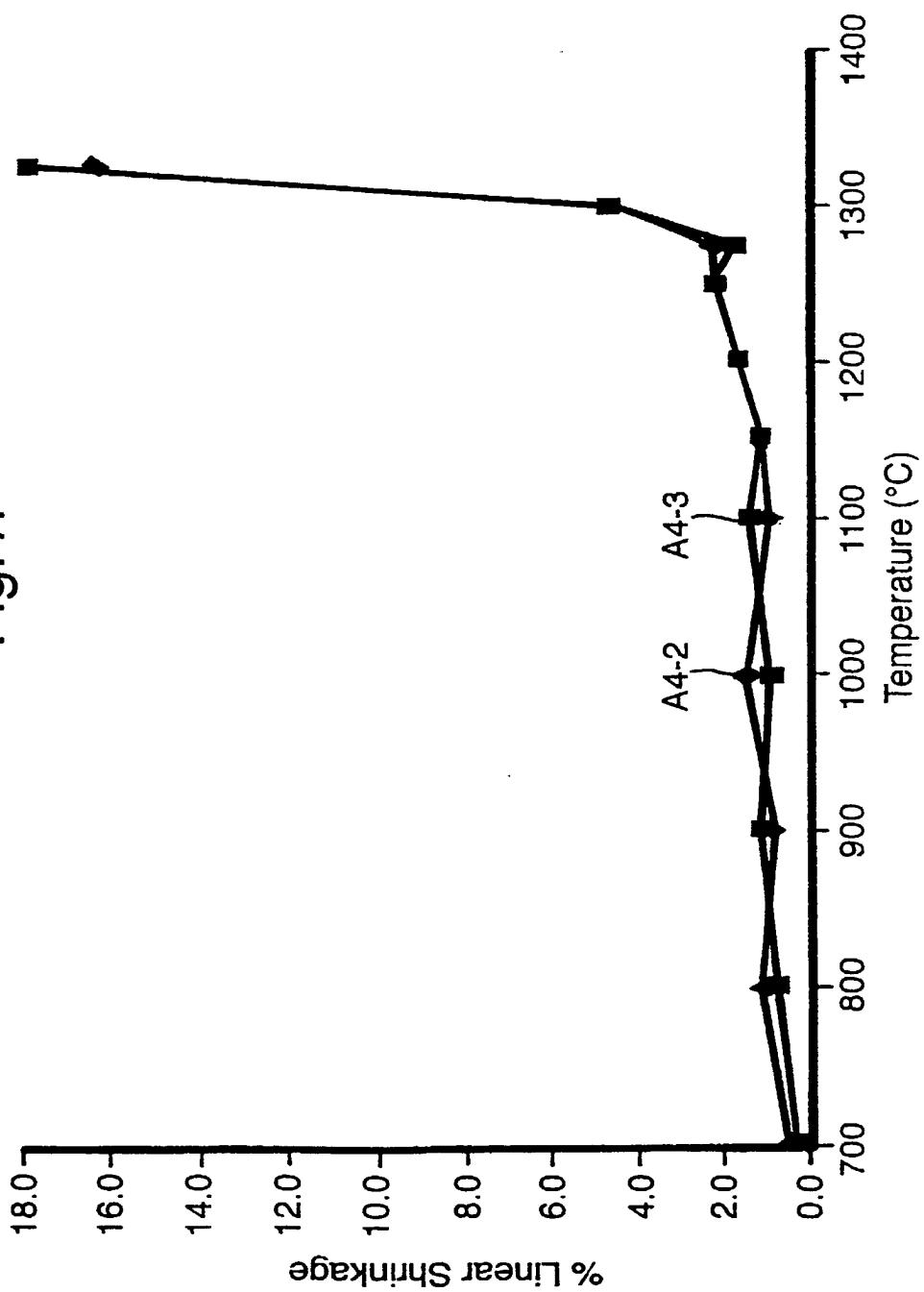
(57) Abstract: A fibre is disclosed having a maximum use temperature of 1200 °C or more which comprises: $\text{SiO}_2 > 64.25$ wt % $\text{CaO} > 18$ wt % $\text{MgO} < 17$ wt % and in which the amount of MgO in mol % is greater than the amount of CaO in mol %. Such fibres have high solubility and low dustiness.

WO 01/19744 A1



For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

Fig. 1.



2/3

Fig. 2.

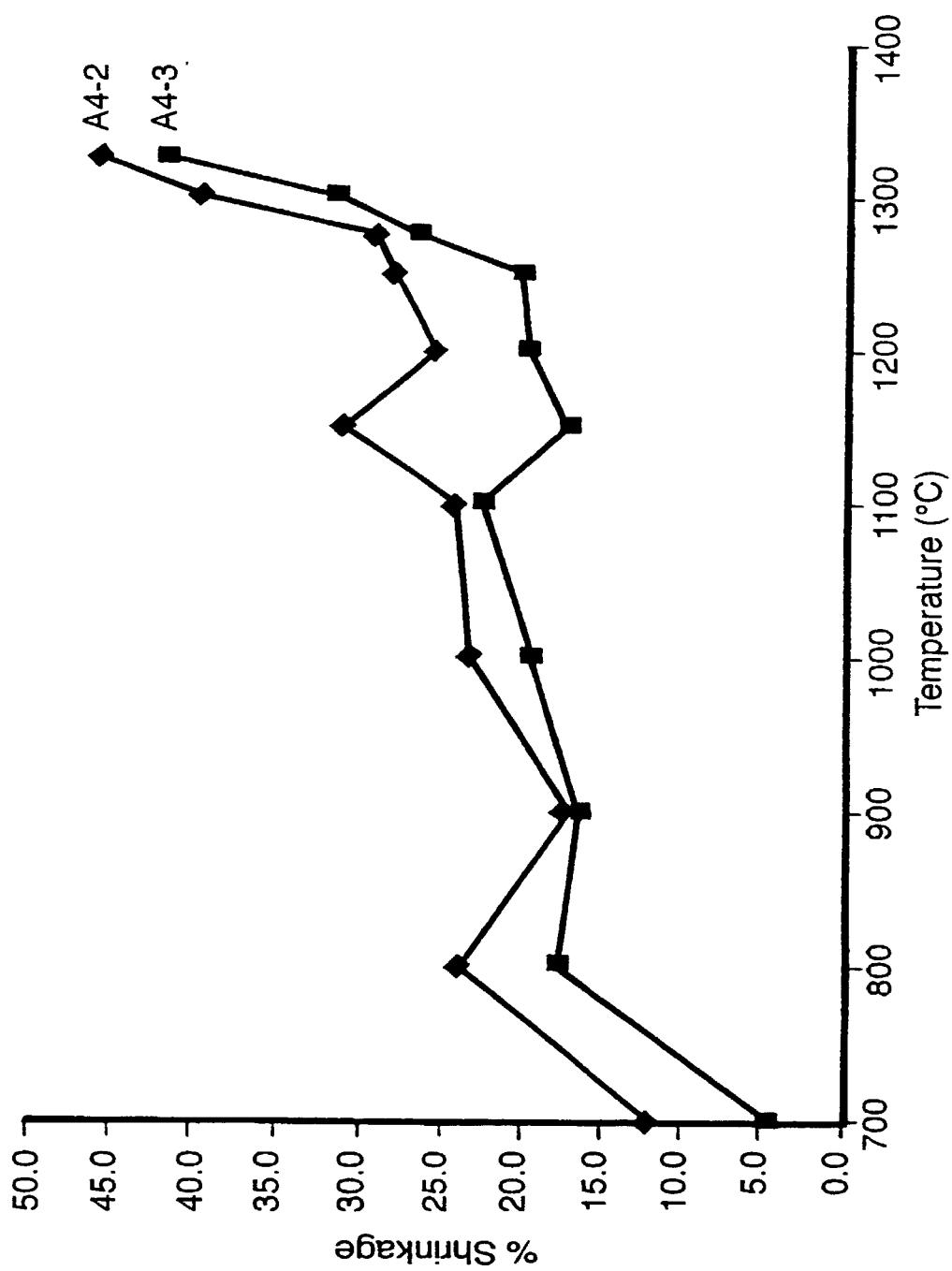
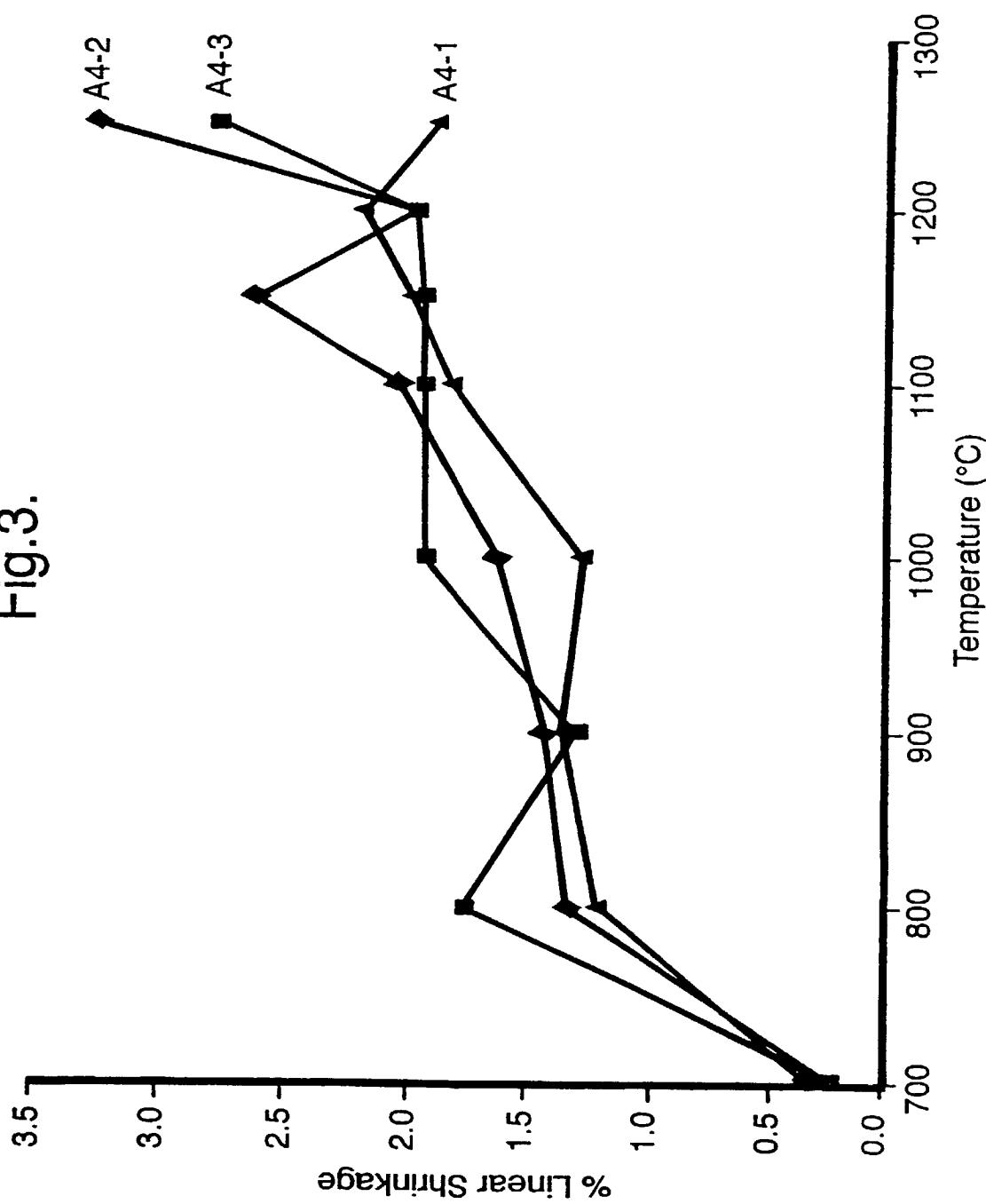


Fig.3.



#5

Attorney Docket No. _____

DECLARATION FOR PATENT APPLICATION

Original

Supplemental

Substitute

PCT

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below), or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

High Temperature Resistant Saline Soluble Fibres
(Title of the Invention)

the specification of which (check one)

is attached hereto

was filed on 24th August 2000 as PCT International Application PCT/GB00/03275

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, § 1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, § 119 (a) - (d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified, by checking the box below, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

| Prior Foreign Applications | | | Priority Claimed | | Copy Attached | |
|----------------------------|---------|-------------------------------------|------------------|----|---------------|----|
| Application Number | Country | Foreign Filing Date (MM/DD/YYYY) | YES | NO | YES | NO |
| 9921504.8 | GB | 10.9.99 | X | | | |
| 9924867.6 | GB | 20.10.99 | X | | | |

I hereby claim the benefit under Title 35, United States Code § 119(e) of any United States provisional application(s) listed below and claim the benefit under Title 35, United States Code, § 120 of any United States application(s), or § 365(c) of any PCT international application(s) designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application(s) in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

| Parent Application Number | Filing Date | Status (Mark Appropriate Column Below) | | |
|---------------------------|-------------|---|---------|-----------|
| | | Patented | Pending | Abandoned |
| | | | | |
| | | | | |

As a named inventor, I hereby revoke all prior powers and appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

69 FIRM NAME: KILPATRICK STOCKTON LLP, 1100 Peachtree Street, Suite 2800, Atlanta, Georgia 30309-4530

| Attorney and/or Agent | Registration No. |
|------------------------|------------------|
| Roger T. Frost | 22,176 |
| Charles Y. Lackey | 22,707 |
| Anthony B. Askew | 24,154 |
| John M. Harrington | 25,592 |
| Robert E. Richards | 29,105 |
| Donald R. Andersen | 28,280 |
| John S. Pratt | 29,476 |
| A. Jose Cortina | 29,733 |
| James L. Ewing, IV | 30,630 |
| Stephen M. Schaetzel | 31,418 |
| James Dean Johnson | 31,771 |
| Charles W. Calkins | 31,814 |
| Larry A. Roberts | 31,871 |
| Jamie L. Greene | 32,467 |
| George T. Marcou | 33,014 |
| Dean W. Russell | 33,452 |
| Richard T. Peterson | 35,320 |
| Charles T. Simmons | 35,359 |
| Tracy W. Druce | 35,493 |
| Eleanor M. Musick | 35,623 |
| Nora M. Tocupis | 35,717 |
| Bruce D. Gray | 35,799 |
| Theodore R. Harper | 35,890 |
| Geoff L. Sutcliffe | 36,348 |
| Leona G. Young | 37,266 |
| David P. Lecroy | 37,869 |
| Suzanne Seavello Shope | 37,933 |
| Mitchell G. Stockwell | 39,389 |
| Jeffery B. Arnold | 39,540 |
| Suil Kang | 39,723 |
| Mary Anthony Merchant | 39,771 |
| Brenda Ozaki Holmes | 40,339 |
| Lisa J. Moyles | 40,737 |
| Michael J. Turton | 40,852 |
| Scott Zimmerman | 41,390 |

| Attorney and/or Agent | Registration No. |
|------------------------|------------------|
| Kimberly J. Prior | 41,483 |
| Alana G. Kriegsman | 41,747 |
| Theodore M. Green | 41,801 |
| J. Steven Gardner | 41,772 |
| Joni Stutman | 42,173 |
| James J. Bindseil | 42,326 |
| Heather D. Carmichael | 42,389 |
| Thomas A. Corrado | 42,439 |
| John K. McDonald | 42,860 |
| Sima Singadia Kulkarni | 43,732 |
| Camilla Camp Williams | 43,992 |
| Christopher J. Chan | 44,070 |
| Li K. Wang | 44,393 |
| John William Ball, Jr. | 44,433 |
| Dawn-Marie Bey | 44,442 |
| Tiep H. Nguyen | 44,465 |
| John M. Briski | 44,562 |
| Michael J. Dimino | 44,657 |
| Kristin L. Johnson | 44,807 |
| J. Jason Link | 44,874 |
| Paul E. Knowlton | 44,842 |
| Bambi F. Walters | 45,197 |
| Cheryl L. Huseman | 45,392 |
| Shelby B. Grier | 45,785 |
| Jennifer R. Seng | 45,851 |
| Vaibhav P. Kadaba | 45,865 |
| Greg Moldafsky | 46,514 |
| J. Michael Boggs | 46,563 |
| Michael K. Dixon | 46,665 |
| Tywanda L. Harris | 46,758 |
| Kristin D. Mallatt | 46,895 |
| Cynthia B. Rothschild | 47,040 |
| John C. Alemanni | 47,384 |
| Geoffrey K. Gavin | 47,591 |
| Janina Malone | 47,768 |
| Robert M. Stevens | 47,972 |

I acknowledge the above-listed attorneys and agents and their firm Kilpatrick Stockton LLP represent my employer (if I am an employee and this application has been or will be assigned to my employer) or the entity with which I have contracted (if I am an

Declaration for Patent Application
Page 3

independent contractor and this application has been or will be assigned to such entity) and in such cases do not represent me individually. I further acknowledge I have not established, nor will I seek to establish, any personal attorney/client relationship with Kilpatrick Stockton LLP in connection with this application and understand that, should I require legal representation, I will obtain such, at my expense, other than through Kilpatrick Stockton LLP.

Send Correspondence to: John S. Pratt, Esq. Customer No. 23370

Kilpatrick Stockton LLP
1100 Peachtree Street, Suite 2800
Atlanta, Georgia 30309-4530



23370

PATENT TRADEMARK OFFICE

Direct telephone calls to: Kristin L. Johnson (404) 815-6389

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

1-00 Full name of first inventor

Gary Anthony JUBB

Inventor's signature

Gary A Jubb

Date 13/3/02

Residence

c/o Morgan Materials Technology Limited, Bewdley Road, Stourport-on-Severn, Worcestershire

DY13 8QR Great Britain GBN

Citizenship

Great Britain

Post Office Address

Same as above

Full name of second inventor

Jean-Louis MARTIN

Inventor's signature

Date

Residence

c/o Thermal Ceramics de France S.A., Lieu dit les Plantees, B.P. 4, F-42680 St. Marcellin-en-Forez,

France

Citizenship

French

Post Office Address

Same as above

Attorney Docket No. _____

DECLARATION FOR PATENT APPLICATION

Original

Supplemental

Substitute

PCT

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below), or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

High Temperature Resistant Saline Soluble Fibres

(Title of the Invention)

the specification of which (check one)

is attached hereto

was filed on 24th August 2000 as PCT International Application PCT/GB00/03275

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, § 1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, § 119 (a) - (d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified, by checking the box below, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

| Prior Foreign Applications | | | Priority Claimed | | Copy Attached | |
|----------------------------|---------|-------------------------------------|------------------|----|---------------|----|
| Application Number | Country | Foreign Filing Date (MM/DD/YYYY) | YES | NO | YES | NO |
| 9921504.8 | GB | 10.9.99 | X | | | |
| 9924867.6 | GB | 20.10.99 | X | | | |

I hereby claim the benefit under Title 35, United States Code § 119(e) of any United States provisional application(s) listed below and claim the benefit under Title 35, United States Code, § 120 of any United States application(s), or § 365(c) of any PCT international application(s) designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application(s) in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

Declaration for Patent Application
Page 2

| Parent Application Number | Filing Date | Status (Mark Appropriate Column Below) | | |
|---------------------------|-------------|---|---------|-----------|
| | | Patented | Pending | Abandoned |
| | | | | |
| | | | | |

As a named inventor, I hereby revoke all prior powers and appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

FIRM NAME: **KILPATRICK STOCKTON LLP**, 1100 Peachtree Street, Suite 2800, Atlanta, Georgia 30309-4530

| Attorney and/or Agent | Registration No. |
|------------------------|------------------|
| Roger T. Frost | 22,176 |
| Charles Y. Lackey | 22,707 |
| Anthony B. Askew | 24,154 |
| John M. Harrington | 25,592 |
| Robert E. Richards | 29,105 |
| Donald R. Andersen | 28,280 |
| John S. Pratt | 29,476 |
| A. Jose Cortina | 29,733 |
| James L. Ewing, IV | 30,630 |
| Stephen M. Schaetzel | 31,418 |
| James Dean Johnson | 31,771 |
| Charles W. Calkins | 31,814 |
| Larry A. Roberts | 31,871 |
| Jamie L. Greene | 32,467 |
| George T. Marcou | 33,014 |
| Dean W. Russell | 33,452 |
| Richard T. Peterson | 35,320 |
| Charles T. Simmons | 35,359 |
| Tracy W. Druce | 35,493 |
| Eleanor M. Musick | 35,623 |
| Nora M. Tocups | 35,717 |
| Bruce D. Gray | 35,799 |
| Theodore R. Harper | 35,890 |
| Geoff L. Sutcliffe | 36,348 |
| Leona G. Young | 37,266 |
| David P. Lecroy | 37,869 |
| Suzanne Seavello Shope | 37,933 |
| Mitchell G. Stockwell | 39,389 |
| Jeffery B. Arnold | 39,540 |
| Suil Kang | 39,723 |
| Mary Anthony Merchant | 39,771 |
| Brenda Ozaki Holmes | 40,339 |
| Lisa J. Moyles | 40,737 |
| Michael J. Turton | 40,852 |
| Scott Zimmerman | 41,390 |

| Attorney and/or Agent | Registration No. |
|------------------------|------------------|
| Kimberly J. Prior | 41,483 |
| Alana G. Kriegsman | 41,747 |
| Theodore M. Green | 41,801 |
| J. Steven Gardner | 41,772 |
| Joni Stutman | 42,173 |
| James J. Bindseil | 42,326 |
| Heather D. Carmichael | 42,389 |
| Thomas A. Corrado | 42,439 |
| John K. McDonald | 42,860 |
| Sima Singadia Kulkarni | 43,732 |
| Camilla Camp Williams | 43,992 |
| Christopher J. Chan | 44,070 |
| Li K. Wang | 44,393 |
| John William Ball, Jr. | 44,433 |
| Dawn-Marie Bey | 44,442 |
| Tiep H. Nguyen | 44,465 |
| John M. Briski | 44,562 |
| Michael J. Dimino | 44,657 |
| Kristin L. Johnson | 44,807 |
| J. Jason Link | 44,874 |
| Paul E. Knowlton | 44,842 |
| Bambi F. Walters | 45,197 |
| Cheryl L. Huseman | 45,392 |
| Shelby B. Grier | 45,785 |
| Jennifer R. Seng | 45,851 |
| Vaibhav P. Kadaba | 45,865 |
| Greg Moldafsky | 46,514 |
| J. Michael Boggs | 46,563 |
| Michael K. Dixon | 46,665 |
| Tywanda L. Harris | 46,758 |
| Kristin D. Mallatt | 46,895 |
| Cynthia B. Rothschild | 47,040 |
| John C. Alemani | 47,384 |
| Geoffrey K. Gavin | 47,591 |
| Janina Malone | 47,768 |
| Robert M. Stevens | 47,972 |

I acknowledge the above-listed attorneys and agents and their firm Kilpatrick Stockton LLP represent my employer (if I am an employee and this application has been or will be assigned to my employer) or the entity with which I have contracted (if I am an

Declaration for Patent Application
Page 3

independent contractor and this application has been or will be assigned to such entity) and in such cases do not represent me individually. I further acknowledge I have not established, nor will I seek to establish, any personal attorney/client relationship with Kilpatrick Stockton LLP in connection with this application and understand that, should I require legal representation, I will obtain such, at my expense, other than through Kilpatrick Stockton LLP.

Send Correspondence to: John S. Pratt, Esq.

Kilpatrick Stockton LLP
1100 Peachtree Street, Suite 2800
Atlanta, Georgia 30309-4530

Customer No. 23370



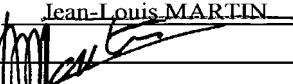
23370

PATENT TRADEMARK OFFICE

Direct telephone calls to: Kristin L. Johnson (404) 815-6389

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of first inventor Gary Anthony JUBB

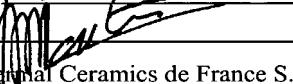
Inventor's signature  Date _____

Residence c/o Morgan Materials Technology Limited, Bewdley Road, Stourport-on-Severn, Worcestershire
DY13 8QR Great Britain

Citizenship Great Britain

Post Office Address Same as above

200 Full name of second inventor Jean-Louis MARTIN

Inventor's signature  Date March 13th, 2001

Residence c/o Thermal Ceramics de France S.A., Lieu dit les Plantees, B.P. 4, F-42680 St. Marcellin-en-Forez,
France

Citizenship French

Post Office Address Same as above